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ON-LINE INFRARED ANALYZER FOR UF_6 IN PURGE CASCADE STREAM

R. E. Barringer

Sponsor: J. G. Million

Analytical Technology Department
Technical Services Division

January 1980

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IN PURGE CASCADE STREAM

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Analytical Technology Department

Technical Services Division

K25RC

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Oak Ridge Gaseous Diffusion Plant
Union Carbide Corporation, Nuclear Division
Oak Ridge, Tennessee

Prepared for the U.S. Department of Energy
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to the public by:

John F. Hester 2/26/96
Asst. Technical Information Officer Date
Oak Ridge K-25 Site

ABSTRACT

An infrared analyzer was applied to the continuous on-stream analysis of uranium hexafluoride in the corrosive gas atmosphere of the Oak Ridge Gaseous Diffusion Plant Purge Cascade. The completed system included continuous display of the infrared analyzer signal on a strip chart recorder in the control room and automatic actuation of the motorized control valves for the Purge Cascade. The UF_6 specific infrared analyzer provided much better control than the ultrasonic acoustic gas analyzers which were previously used and were non-specific in response.

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SUMMARY

An infrared analyzer was applied to the continuous analysis for UF_6 in the K-402-9 Purge Cascade process gas stream replacing the ultrasonic acoustic gas analyzers. A newly designed recorder bias and input inverter permitted direct coupling of the infrared analyzer to the existing automatic control system. Continuous display of the infrared analyzer signal on a strip chart recorder in the central control room and actuation by the infrared analyzer signal of motorized control valves in the Purge Cascade completed the system. The UF_6 specific infrared analyzer provided better control and consequently a more stable Purge Cascade than the previously used acoustic gas analyzers which were subject to interference from components other than UF_6 in the process gas stream. The initial infrared installation operated almost continuously for 10 months with only minor adjustments before the infrared sample cell became unusable.

INTRODUCTION

Stable operation of the Gaseous Diffusion Purge Cascade depends on control of the uranium hexafluoride (UF_6) concentration in the cascade gas stream. The acoustic gas analyzer (AGA), traditionally used for on-stream UF_6 analysis, operates through the reduction in the speed of ultrasonic sound waves passing through gas samples and is therefore non-specific but senses all gases in proportion to their molecular weight. Gases other than UF_6 are sensed proportionally as UF_6 by the acoustic gas analyzer with resultant inefficient operational control of the purge. Refrigerant-114 (R-114) and chlorine trifluoride (ClF_3), found in the process stream, are detected by the acoustic gas analyzer as a proportional amount of UF_6 , and erroneous signals are sent to the automatic controls of the purge cascade. Additional problems with the acoustic gas analyzers are maintenance due to age and unavailability of parts.

Criteria for a replacement analyzer for this application were that the analyzer be specific for UF_6 , stable in operation, and capable of continuous operation.

Possible specific analyzers include automatic mass spectrometers, on-line infrared analyzers, and ultraviolet spectrophotometric analyzers. Of these choices, the infrared analyzers appeared reasonably economical and easy to install.

EXPERIMENTAL

FEASIBILITY OF THE INFRARED METHOD

Although UF_6 is infrared active with a strong absorption at 625 wavenumbers ($16.0\ \mu\text{m}$), the feasibility of the infrared analysis in the mixed gases of the Purge Cascade stream was not fully known. Process gas from the Purge Cascade representative of that expected at the final sampling point was passed through an infrared analyzer cell. The spectrum obtained is shown in Figure 1. Adequate separation of the UF_6 infrared absorption from other components indicated the infrared approach was a viable one.

EQUIPMENT

Infrared Spectrophotometer

The infrared instrument available was a laboratory-type Beckman AccuLab 6. Experience had been gained with this instrument in sampling various gas streams in the Purge Cascade and other systems at the Oak Ridge Gaseous Diffusion Plant (ORGDP). The selectivity and sensitivity of this infrared instrument appeared adequate. The AccuLab 6 could be operated in a fixed wavelength mode for continuous monitoring of any chosen absorption peak. A provision for purging the monochromator with dry air reduced in-leakage of wet or possibly corrosive atmospheres into the infrared instrument. The capability of the AccuLab 6 to scan the infrared spectrum over the range from 4000 to $250\ \text{cm}^{-1}$ permitted surveying components for gases other than UF_6 at an equipment cost that was not much greater than for a fixed-wavelength infrared analyzer. An unknown factor was the continuous-duty performance of the AccuLab 6 spectrophotometer in the hot atmosphere of the Purge Cascade. All indications were that the AccuLab 6 infrared spectrophotometer would be a suitable basic instrument for a UF_6 infrared analyzer.

Infrared Sample Cell

The infrared sample cell was of the flow-through type. Silver chloride was used as windows for both its transmission of infrared energy covering the range of 4000 to 400 wavenumbers and its resistance to corrosive gases in the purge cascade, e.g., UF_6 and ClF_3 . At the gas pressures and UF_6 concentration existing at the sampling point, the optimum cell pathlength was calculated as a few millimeters. An existing 6-mm pathlength cell with a corrosion-resistant monel body was used in the initial phases of the experiment. Later, an improved cell of 7-mm pathlength was designed and fabricated. The 7-mm cell parts contacting the silver chloride windows were gold plated to reduce corrosion by silver chloride which reacts with most metals, particularly in moist atmospheres. The 7-mm cell is shown in Figure 2. Details of the cell construction are shown in the Appendix (Figures A-1 and A-2).

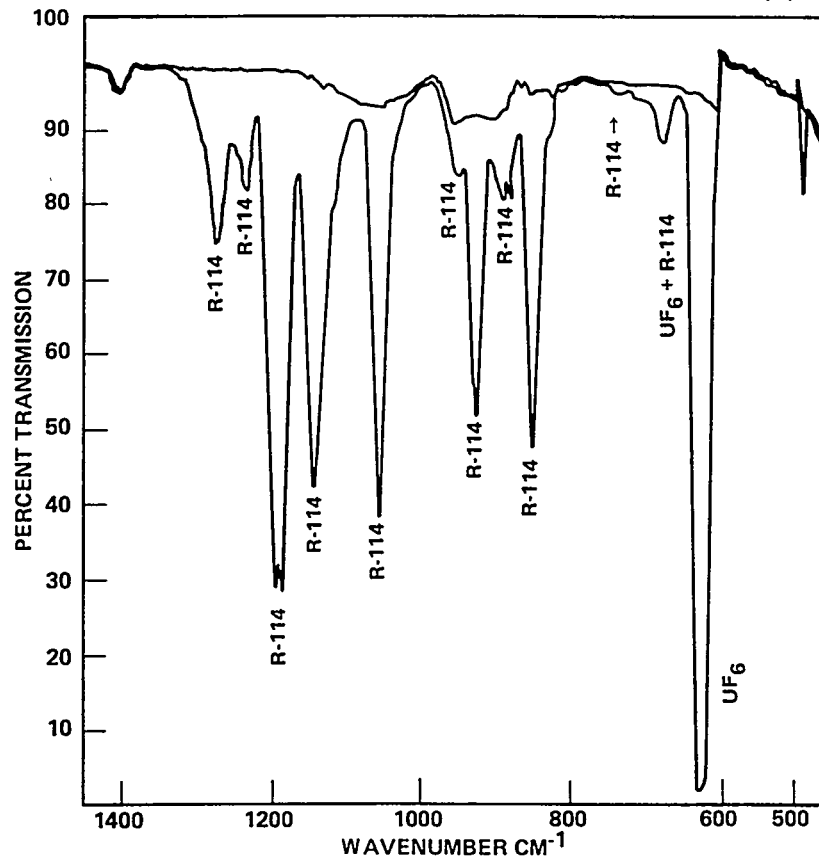


Figure 1
PARTIAL INFRARED SPECTRUM OF PURGE CASCADE STREAM

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(U)

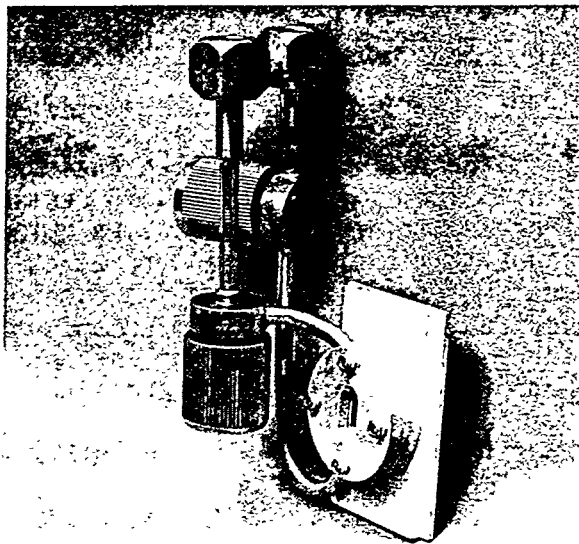


Figure 2
7mm CORROSION RESISTANT INFRARED GAS CELL

Reference Beam Attenuator

A beam attenuator of the venetian blind type with screw adjustment was placed in the reference beam of the spectrophotometer. The attenuator reduced the reference beam transmission to balance it with the sample beam transmission through the infrared cell under blank conditions with no UF_6 present. More attenuation of the reference beam was required as the windows of the infrared cell became clouded during the course of time. The screw adjustment held the attenuator constant under the vibration experienced in the Purge Cascade.

Sampling Manifold

A sampling manifold provided control of the gas flow from the sampling port of the Purge Cascade through the infrared sample cell plus connections for other services such as vacuum, air, etc., as needed. The sampling manifold was an adaptation of one designed by R. L. Farrar, Jr.¹ A detailed drawing of the sampling manifold is in the Appendix (Figure A-3).

Materials for the manifold valves, tubing, and fittings were monel, stainless steel, or fluorocarbon polymer for corrosion resistance to fluoride atmospheres. A small bellows pump with stainless steel and fluorocarbon polymer parts exposed to the sample gas (Metbellows MB-41) provided circulation of the sample gas within or through the manifold and infrared cell as needed.

Vacuum Pump and Chemical Trap

Evacuation of the Sampling manifold-infrared cell system was by vacuum pump (Welch Duo Seal). Protection for the vacuum pump from UF_6 and other corrosive gases was provided by a chemical absorption trap filled with alternate 1/2-in. layers of soda-lime and alumina (~ 8 mesh). The absorber filled a 3-in. diameter steel cylinder to a depth of 33 in.

Recorder Bias and Input Inverter

The output signal from the AccuLab 6 infrared analyzer increased from 0 to 1 volt with decreasing percent transmission (increasing absorbance), i.e., with increasing UF_6 concentration. The existing automatic control system used with the old acoustic gas analyzer and to which the UF_6 infrared analyzer was to be connected operated with decreasing signal voltage for increasing UF_6 concentration. To make the infrared analyzer compatible with the existing control system, an analog bias and input inverter was designed and fabricated which produced a decreasing output voltage with increasing input voltage, i.e., increasing UF_6 concentration. Figures 3, 4, and 5 show the construction of this device which was called a Recorder Inverter, since it could readily shift and invert an analog input signal to a chart recorder or device with similar input characteristics. Construction details and parts list are found in the Appendix (Figure A-4 and Table A-1).



Figure 3
RECORDER BIAS AND INPUT INVERTER — FRONT

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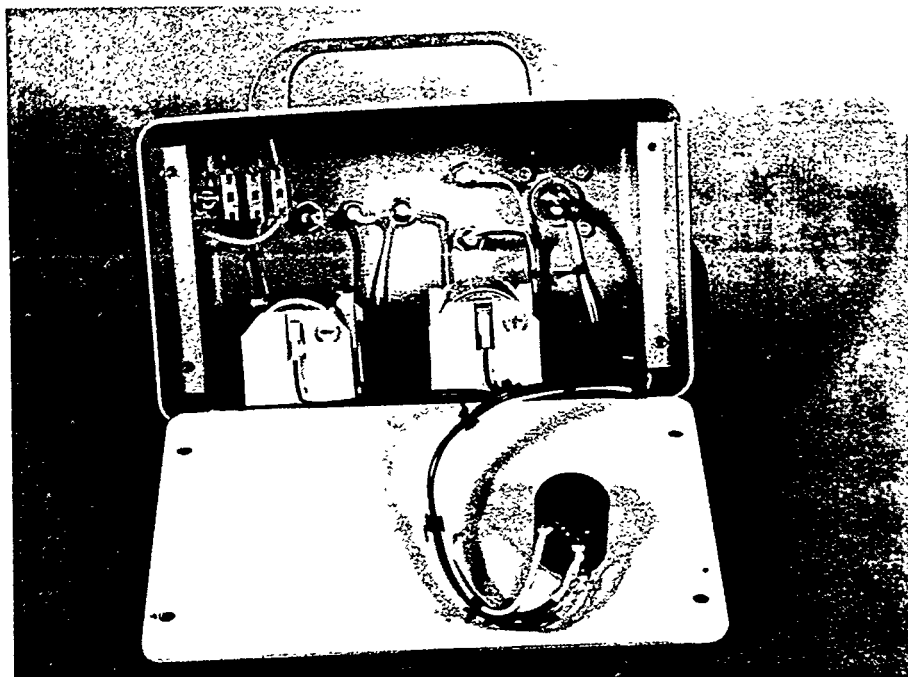


Figure 4
RECORDER BIAS AND INPUT INVERTER — INSIDE FROM FRONT

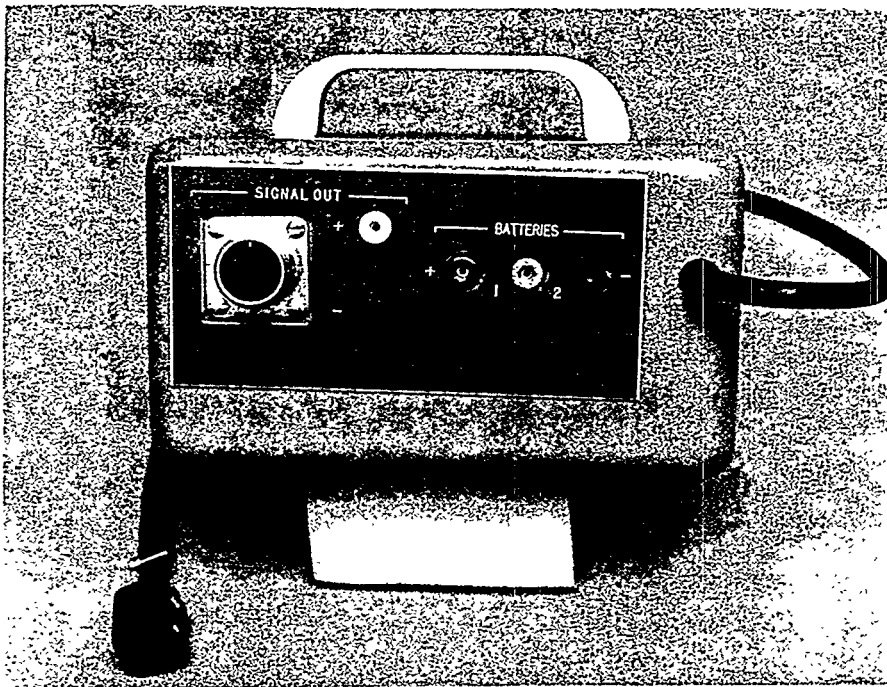


Figure 5
RECORDER BIAS AND INPUT INVERTER — REAR

Signal Converter

The voltage output signal from the Recorder Inverter was converted to a 4- to 20-milliamperes DC current signal by a signal converter (Transmation 230 IT). The 4- to 20-milliampere signal activated automatic controls in the central control room of the Purge Cascade and also produced a continuous trace on a strip chart recorder for the control room operators reference. Motorized valves vented air and other light gases from the Purge Cascade in response to automatic controls, thus maintaining a constant UF_6 concentration at the process stream sampling point.

Completed Infrared Analyzer Assembly

The equipment described above was assembled and interconnected into an operating infrared analyzer system. Figure 6 is a block diagram of the completed system as installed. Figures 7 and 8 show the completed installation in K-402-9 Purge Cascade. The infrared analyzer was installed near the existing acoustic gas analyzers and used a common line for sampling the Purge Cascade stream. Tracing the sample lines and sampling manifold with a self-limiting heating tape (Auto-Trace 10 PTV-1 by Chemelex) reduced condensation of UF_6 in the system. A maximum sample line temperature of approximately 150°F was reached by this tape when connected directly to 120-volt AC power.

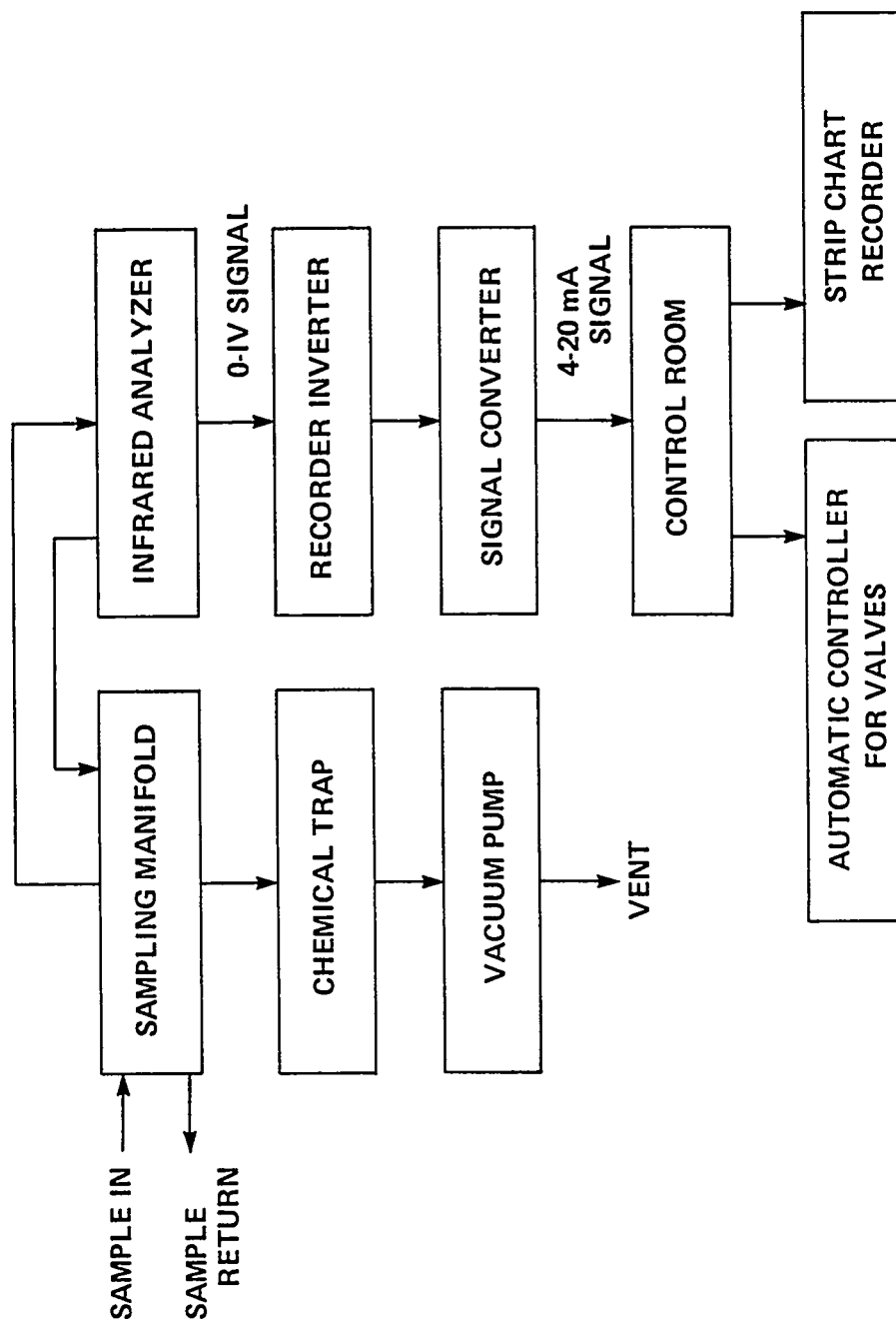


Figure 6
DIAGRAM OF INFRARED ANALYZER INSTALLATION

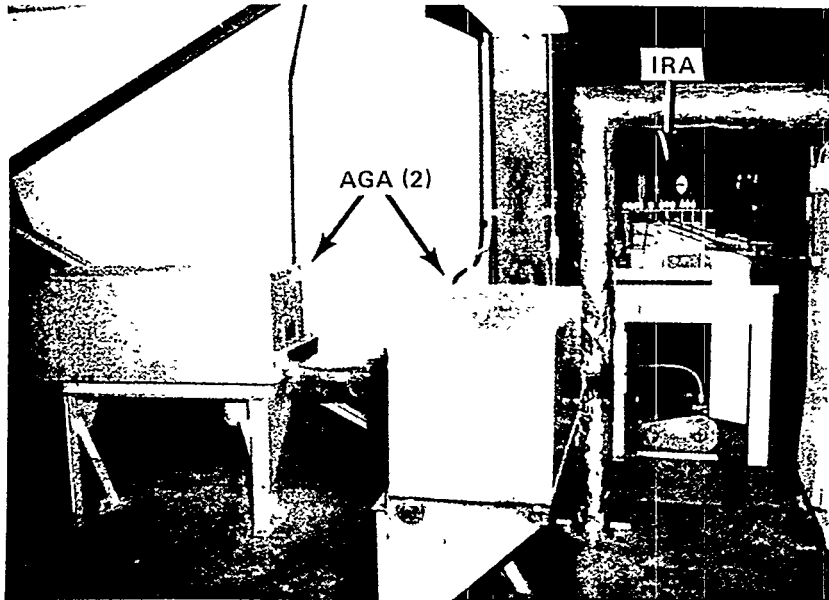


Figure 7
INFRARED ANALYZER INSTALLATION SHOWING
ACOUSTIC GAS ANALYZERS

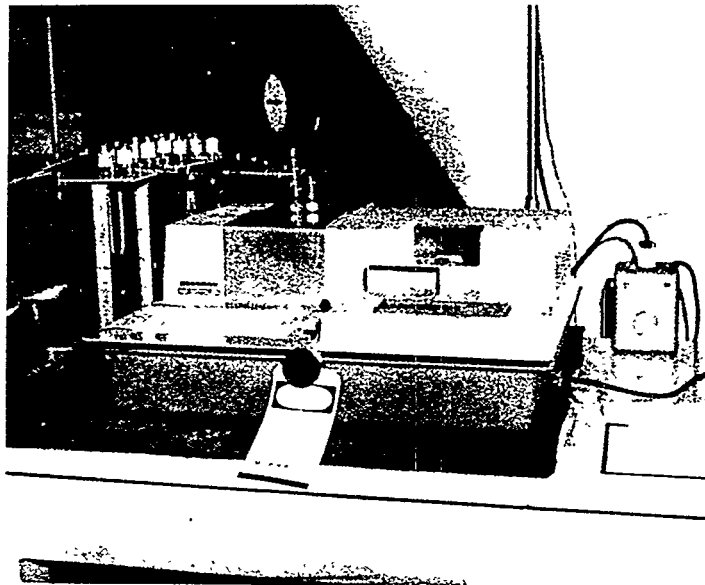


Figure 8
INFRARED ANALYZER INSTALLATION SHOWING
SAMPLING MANIFOLD

RESULTS

CALIBRATION OF INFRARED CELL FOR UF_6

The existing 6-mm pathlength infrared cell was calibrated by admitting known pressures of UF_6 into the cell and noting the percent transmission recorded on the AccuLab 6 at the 625 cm^{-1} infrared absorption band of UF_6 . The calibration data is given in Table 1. Converting the percent transmission to absorbance [$A = \log(1/\%T \times 100)$] and plotting the net absorbance difference from the blank against the UF_6 pressure showed a linear relationship. A linear regression plot of the calibration data is shown in Figure 9.

OPERATING EXPERIENCE WITH INFRARED ANALYZER

The infrared analyzer was installed on May 5, 1977, and the output signal connected to the automatic Purge Cascade controls. Some adjustment in response of the automatic controller was required to match the characteristics of the infrared analyzer rather than those of the acoustic gas analyzer.

Simultaneous recordings were made of the signals over a period of several days from the infrared analyzer and the acoustic gas analyzer which was sampling the same Purge Cascade gas stream. One 13-hr period of both recorder charts is shown in Figure 10. Time progresses from top to bottom of the charts while UF_6 concentration increases are shown from left to right of the charts. As shown, the infrared UF_6 response held a course lying along the center point as set by the controller. On the other hand, the acoustic gas analyzer showed a gradual increase in UF_6 concentration followed by a decrease in UF_6 concentration, and finally, a widely oscillating period ending in a midpoint UF_6 concentration.

From other analyses, it was known that refrigerant-114 was gradually accumulating during the first 6 hr of the recording. During the next 3 to 4 hr, the excess refrigerant-114 was withdrawn from the Purge Cascade. Finally, a surge of impurities such as air and/or chlorine trifluoride from downstream in the cascade caused an oscillating response as the automatic controls opened or closed valves in an attempt to pass the impurities while maintaining a constant UF_6 concentration. After the large surge passed, the Purge Cascade once again settled to a steady operating condition. This comparison shows that the infrared analyzer response was little affected by changes in the refrigerant-114 concentration or a surge of impurities in the cascade.

It soon became apparent that UF_6 concentration was not the only criterion for optimum operation of the Purge Cascade. Electrical loads on the compressors became excessive whenever the concentration of the heavier impurities, such as chlorine trifluoride or refrigerant-114, rose significantly. In these cases, the automatic controller was reset to maintain a lower-

Table 1
CALIBRATION OF 6MM INFRARED CELL

<u>Pressure of UF₆ (mm Hg)</u>	<u>Absorbance</u>
0	0
3.0	0.182
5.0	0.301
7.0	0.423
10.0	0.594

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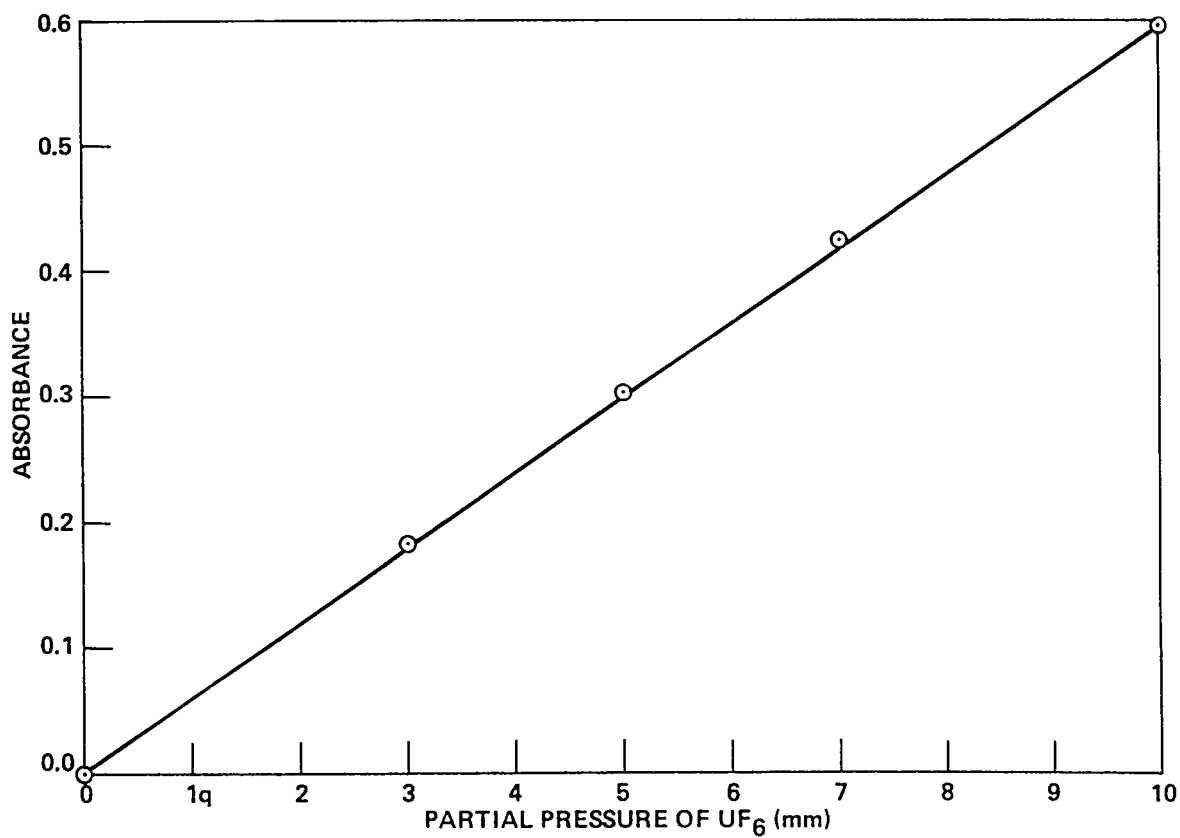


Figure 9
CALIBRATION OF 6mm INFRARED CELL

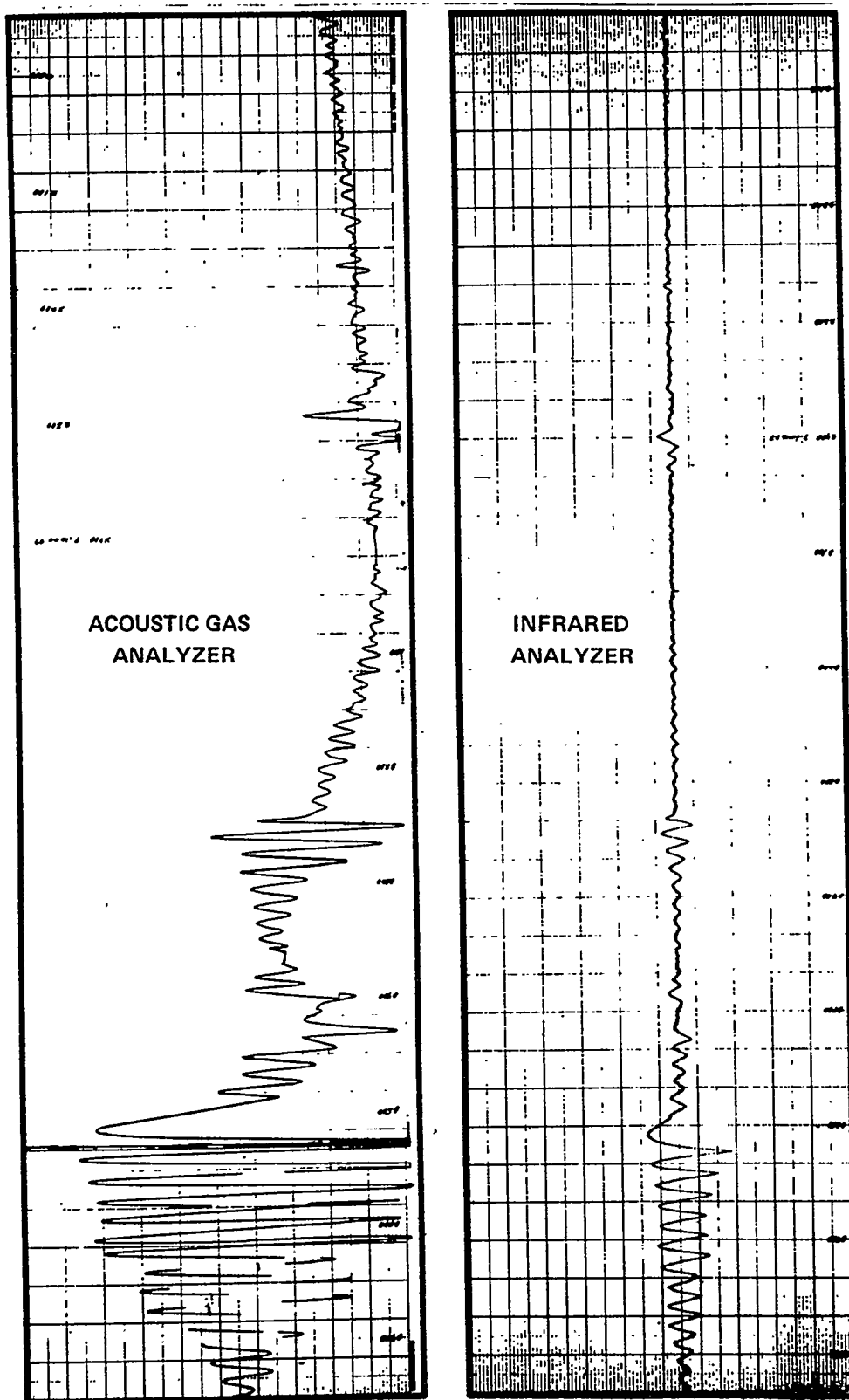


Figure 10
SIMULTANEOUS 13-HOUR ACOUSTIC GAS ANALYZER AND INFRARED
ANALYZER CHARTS OF PURGE CASCADE STREAM

than-normal UF_6 concentration until the excess of heavier impurities passed through or were removed from the Purge Cascade.

Based on the above experience, it was not necessary to calibrate accurately the new 7-mm infrared cell for UF_6 . Sufficiently close control was achieved by a good approximation based on the 6-mm cell calibration. The relative changes in UF_6 concentration coupled with motor load changes provided the necessary references for control of the Purge Cascade.

The control room operators soon learned that the acoustic gas analyzer response served as an alert to changing impurity levels or impurity surges in the cascade stream. Rapidly changing conditions often required manual assistance to the automatic controls to achieve some stability in the Purge Cascade during these periods.

Continuous quantitative infrared measurements of a gas require that the pressure remain constant or be a known value and entered into the calculations. In the infrared analyzer installation, the pressure was held to a degree of constancy by a bellows pump which circulated the process gas stream to the sampling point. An average pressure of 4.2 in. (107 mm) of mercury absolute was noted over a 6-month period with variations, generally $\pm 10\%$. The infrared response, likewise, varied 10% with the pressure changes. In practice, this proved adequate for control purposes and permitted stable operation of the Purge Cascade.

Other factors such as loading of the compressor motors by refrigerant-114 of chlorine trifluoride did not always permit operating the Purge Cascade at a constant UF_6 concentration until these impurities had passed out of the Purge Cascade.

MAINTENANCE EXPERIENCE

The initial infrared analyzer that was installed operated continuously for 10 months with only a brief shutdown when an infrared source lamp burned out. Ultimately, the silver chloride infrared cell windows became coated and opaque. This problem, not yet completely resolved, is a disadvantage to on-stream analysis techniques. Periodic replacement of short, well-heated sample lines and infrared sample cells is currently the best solution.

QUALITY ASSURANCE CONTROL

A simple method was devised to determine whether the infrared analyzer was operating properly. As the cell windows gradually became clouded, the background absorption level seen by the infrared analyzer became greater. To compensate for the drift, the analyzer was readjusted such that a blank gas sample showed zero absorbance (100% transmission). Either dry plant air or a vacuum was found adequate for a blank gas. The blank analyzer setting was made by adjusting the reference beam attenuator until

both the sample and reference beams balanced at 100% transmission on the spectrophotometer recorder chart. This setting was performed at the 625-cm^{-1} absorption band of UF_6 .

After zero readjustment, the response of the infrared analyzer to an absorbing material was tested for accuracy. A simple method for testing was devised involving a wire mesh screen (14 mesh nickel was readily available to author). The wire mesh screen was read as 50.5% transmission on a more expensive laboratory infrared spectrophotometer. Response of the infrared analyzer between 49% and 52% transmission was considered an adequately accurate response.

One precaution must be observed in the use of the wire mesh screen. If the screen is placed in the normal sample holder position, i.e., at the point of the smallest beam cross section, an individual vertical wire can block a disproportionate share of the sample light beam. One solution to this problem is to hold the wire screen against the opening in the analyzer monochromator housing where the AccuLab 6 light beam is large and not sensitive to individual wires in the screen. A test for non-interference of individual wires is to slowly move the screen laterally and vertically before the lamp house opening. If no change in response is evident, individual wires are not detected. A screen used in the above manner permits checking the infrared analyzer on location without removing the sample cell.

Experience showed that a weekly adjustment of the zero absorbance (100%T) setting and response check as above was generally adequate.

CONCLUSIONS

The infrared analyzer is a practical method of on-line analysis for UF_6 in the mixed gases found in the Purge Cascade. The instrumentation is moderate in cost and had been found to be dependable in continuous operations.

Advantages that are realized over the acoustic gas analyzer include (1) more stable operating conditions for the Purge Cascade due to insensitivity of the infrared analyzer to other components found in the gas stream, (2) unattended automatic control of the Purge Cascade for periods of several hours, and (3) more efficient purging of impurity components from the gas stream due to stable operating conditions. Larger amounts of gases are separated in the Purge Cascade than operating personnel believed possible from past experience.

REFERENCES

1. Farrar, R. L., Jr.; Enrichment Technology Division, Union Carbide Corporation, Nuclear Division, Oak Ridge Gaseous Diffusion Plant, Oak Ridge, Tennessee; *Private Communication*.

ACKNOWLEDGMENTS

A. C. Jordan and B. W. Tomlinson of the Operations Division requested an on-stream analyzer for UF_6 analysis and provided needed information and support for the installation and operation of the infrared analyzer.

R. L. Farrar, Jr. of the Enrichment Technology Division provided information and drawings of chemical traps and sampling manifold based on his experience with the corrosive gas streams of the Oak Ridge Gaseous Diffusion Plant. W. G. Schwab of the Enrichment Technology Division provided the 6-mm gas cell used initially and obtained the calibration data for UF_6 .

APPENDIX

DETAILS OF EQUIPMENT CONSTRUCTION



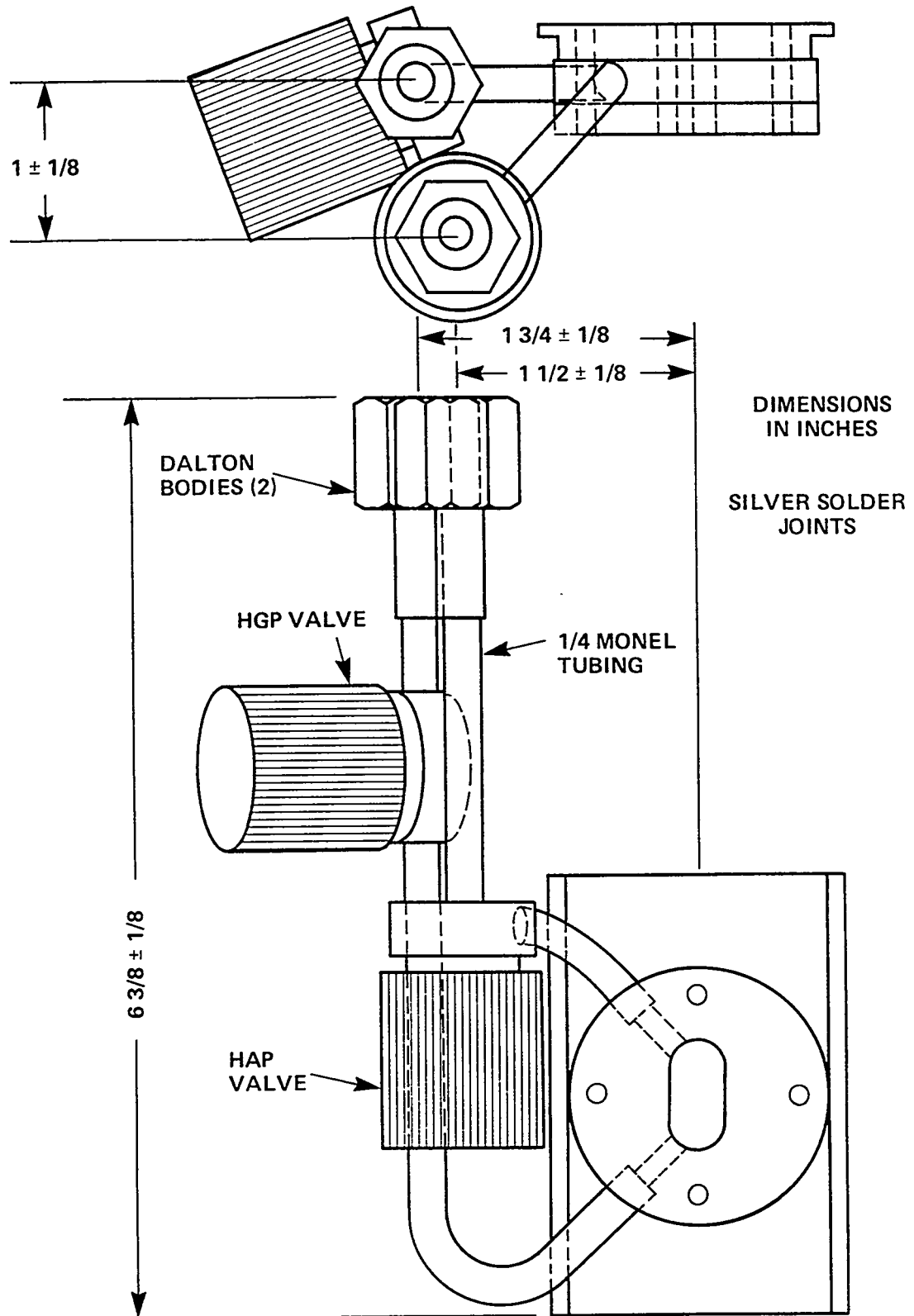
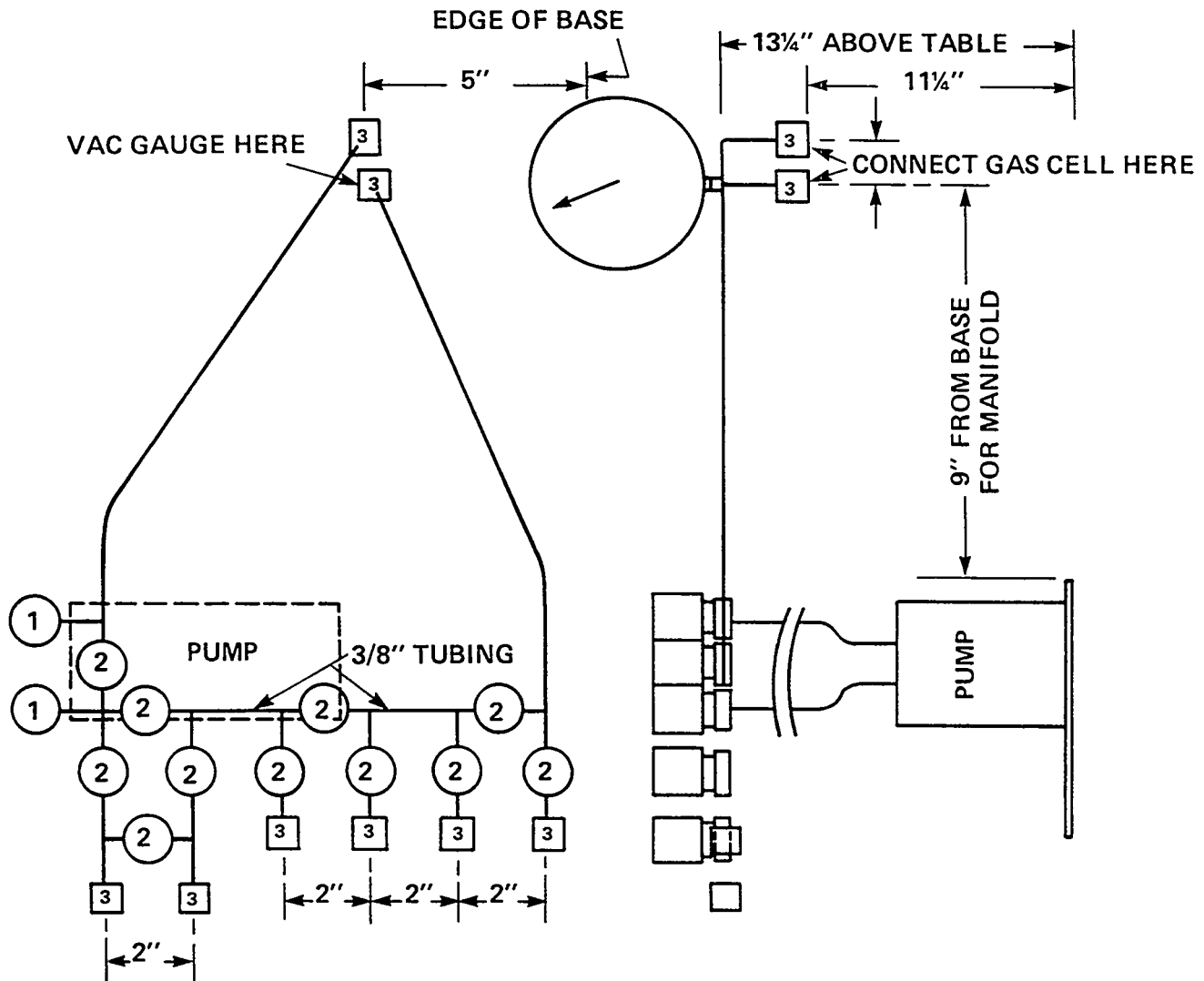


Figure A-2
FINAL ASSEMBLY OF 7mm CORROSION-RESISTANT GAS CELL

DWG. NO. K/G-79-1485
(U)



1 - HAP VALVE
2 - HGP VALVE
3 - DALTON BODY 76-594-0340
PUMP: METBELLOWS MB-41
07-302-0350
GAUGE: VACUUM 4 1/2" 06-034-0270

SCALE: 1/4" = 1"
MATERIAL: MONEL TUBING, 1/4"
OD EXCEPT MANIFOLD WHICH
IS FABRICATED FROM 3/8" &
1/4" TUBING

Figure A-3
SAMPLING MANIFOLD FOR INFRARED ANALYZER

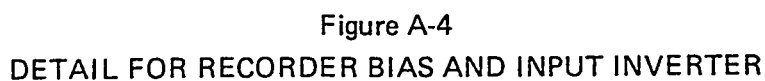


Table A-1
PARTS LIST FOR RECORDER BIAS AND INPUT INVERTER

<u>Quantity</u>	<u>Item</u>	<u>UCC Stores Number</u>
2	Battery, Mercury RM 42R	06-770-0481
2	Holder, Battery	06-770-1551
1	Cabinet, instrument	06-776-0547
1	Clamp, Cable, MS connector 14S	06-843-0131
2	Jack, phone tip, Black	06-850-2704
1	Jack, Phone tip, Orange	06-850-2784
1	Jack, phone tip, Red	06-850-2804
1	Jack, phone tip, White	06-850-2824
1	Socket, Cable - Cinch Jones S-304-CCT	06-860-1029
1	Insert, MS connector 14S-IP	06-868-1584
1	Insert, MS connector 14S-1S	06-868-1604
1	Shell, MS connector 3102A-14S(639)	06-868-4164
1	Shell, MS connector 3106A-14S	06-868-4444
1	Dial, turns-counting Bechman RB	06-881-3802
1	Potentiometer 20,000 Ω 10 turn Bourns 3500S-2-203	06-930-1135

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